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Article

Environmental Method for Synthesizing Amorphous Silica Oxide Nanoparticles from a Natural Material

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Abstract: Numerous studies have been performed on the generation of several silicon-based engineering materials that often have used chemical materials that have high risks for health and the safety of the environment. Generally, in the synthesis of Nano-silica, tetramethoxysilane, tetraethoxysilane, and tetraethyl orthosilicate (TEOS) are used as precursor materials; however, these materials are toxic and expensive for the production of Nano-silica. This paper presents an environmentally friendly short method (EFSM) with high efficiency for the synthesis of amorphous silica oxide Nanoparticles by using agricultural waste called rice husks (RHs). Use of the EFSM method as an alternative to the chemical methods would have the advantages of fast and simple operation, controllability, great pureness of the Nanoparticles, and low manufacturing cost. A Nanoparticles (NPs) evaluation was conducted with energy-dispersive spectroscopy (EDS), field emission scanning electron microscope (FESEM) and X-ray fluorescence (XRF). By applying the EFSM method, non-toxic amorphous silica nanoparticles with a purity of 94.5% and particle size less than 100 nm was synthesized without using any chemical material.

Keywords: environmentally friendly short method; rice husks; amorphous silica Nanoparticle; non-toxic

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1. Introduction

For the synthesis of silica-based materials and its application in various industries, there are different methods utilized, such as the sol-gel method [1–4], chemical vapor deposition phase (CVD) [5], micro-emulsion process [6], combustion synthesis [7], and microwave method [8]. Most of these techniques are complicated, and too much energy and time will be consumed for production.

At the industrial scale, silica is traditionally synthesized in such a way that sodium carbonate powder with quartz sand is placed at high temperature (about 1300 °C) to produce silicate sodium powder, followed by silicate sodium reacted with sulfuric acid for producing silica [9]. The traditional method for producing 1 ton of silica uses 0.53 tons of sodium carbonate, and 0.51 tons of sulfuric acid as feed. During this process, 0.23 tons of carbon dioxide, 0.74 tons of sodium sulfate, and 20 tons of effluents will be generated. Due to the widespread use of silica in various industries, its production has increased in the coming decades as this traditional method will cause severe problems for the environment in the future [10–15]. Recently, much attention has been paid to the use of plant pre-materials for the synthesis of Nanoparticles as an eco-friendly method due to its advantages over chemical methods. The benefits of this method are its availability, the low cost of plant materials, the elimination of chemical substances and the reduction of energy consumption [16].

Rice husk contains 20% and 33% of the paddy weight with an annual global amount of 137 M tons [17]. Regarding the massive amount of annual rice husks (RHs) production, the use of RHs has had low value associated with it, due to their low nutritional value and tough structure [18,19]. It has been recycled only for low-value agricultural applications. According to previous studies, it is anticipated that total rice consumption will have reached 450 M tons (milled basis) in the year 2020. It has increased about 6.6% compared to the year 2007 [20]. As a result, a massive amount of rice husk is produced which can be used in the industry to solve the problem of rice husk removal and prevent environmental pollution [21]. Recently, many rice mills have utilized RHs to generate energy for the grinding process and for some home lighting in rural areas. Burning RHs produces rice husk ash (RHA), which is known as a biochar substance, i.e., a toxic organic pollutant that is dangerous to the ecosystem and social health. Studies show that one of the common disposal systems is called open-field burning. This causes greenhouse gas emission, air-polluting, and energy loss. Disposing only a small amount of RHA in open or underground areas can have hazardous effects on human health and the environment [22]. RH could be a suitable candidate for silica-based materials because of its special silica content (15−28 wt %, dependent on the type, weather, and geographic situation) and abundant availability [23–25].

RH has large amounts of silica, which can achieve nontoxic amorphous nanoparticles. These nanoparticles have a high specific surface area and are eco-friendly compared to the crystalline state which is classified as a carcinogenic substance [26,27]. Due to the intricate and time-consuming nature of the synthesis method of silica nanoparticles, which are obtained by conventional methods such as sol-gel, vapor phase chemical deposition, its widespread applications were restricted [27,28]. Nevertheless, by employing a simple method introduced in this article, RHs can be used as an economical source for the production of non-toxic amorphous biogenic silica nanoparticles that eliminates the problems associated with waste RHs and its environmental pollution [29].

2. Materials and Methods

In this study, a planetary ball mill apparatus and thermal decomposition technique (calcination) were used to synthesize amorphous silicon dioxide (SiO_2) Nanoparticles.

2.1. Synthesis of NPs

To increase the purity of Nanoparticles, the used RHs need to be dust-free. Therefore, the RHs were washed with water and then placed in the oven at 120 °C for one day to dry thoroughly [30–32].

2.1.1. RHs Ball Milling

To increase the RHs special surface area (SSA), RHs must be milled by using a planetary ball mill apparatus to pass through mesh No 70 [28]. Its process is shown in Figure 1. Increasing the SSA results in the improvement of heat absorption by the RHs during the thermal decomposition step. Moreover, it raises the breaking of chemical bonds, which causes the promotion of the efficiency and purity of the Nanoparticles. The ball mill conditions have been done with 10 numbers of the ball in 3 steps and 4-time intervals of 15 min with different rpm, which are shown in Table 1.

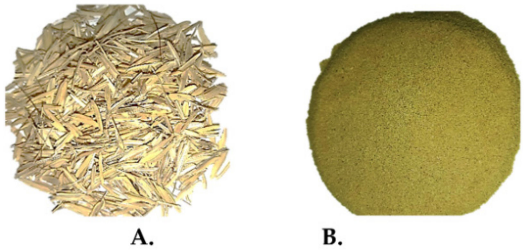

Figure 1. (**A**) Rice husks (RHs); (**B**) ball-milled RHs.

Table 1. Ball mill condition.

	One Time Duration	Two Time Duration	Three Time Duration	Four Time Duration	Five Time Duration
One step (rpm)	300	400	500	600	700
Two step (rpm)	400	500	600	700	700
Three step (rpm)	500	600	700	700	700

2.1.2. Optical Microscope Images

Optical microscopy was used to image the ball-milled RHs. The related image is shown in Figure 2.

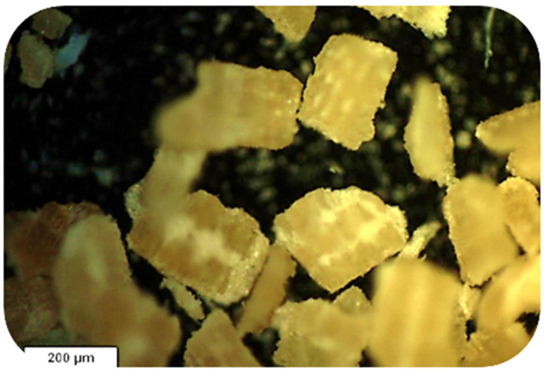

Figure 2. Low magnification optical microscopy image from RHs ball-milled.

Figure 2, shows that the powdered RHs of about 200 microns were obtained. As shown in Figure 3, the magnification of the optical microscope shows that the silica particles are separated from the structure of the RHs.

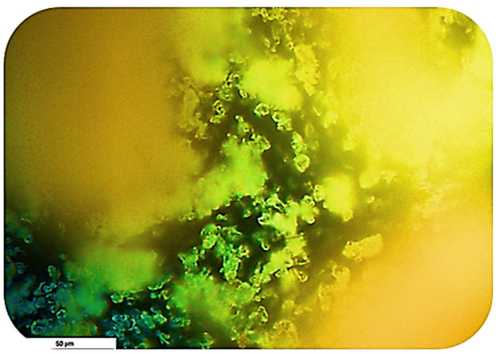

Figure 3. High magnification optical microscope image of RHs ball-milled.

2.1.3. Thermal Decomposition Step

At this stage, the thermal decomposition of the ball-milled material is conducted by placing it in the furnace for five h at 600 °C with a heating rate of 10 °C/min to remove the organic components [33,34]. It is shown in Figure 4.

A. B.

Figure 4. Ball milled RHs (**A**) before and (**B**) after the calcination process. Silicon oxide nanoparticles (NPs) were formed upon calcination.

3. Results and Discussions

Non-conductive materials need to be coated with a conductive material before proceeding to take an image. For this purpose, the synthesized silica Nanoparticles are covered with gold and then scanned. The field emission scanning electron microscope (FESEM) instrument model MIRA3-TESCAN-XMU was used to study the morphology of the NPs synthesized. The results of the FESEM test on the synthesized NPs are shown in the Figure 5.

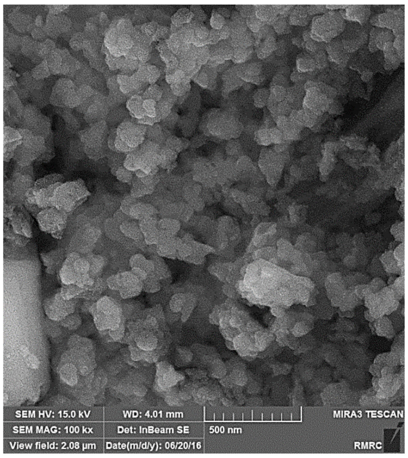

Figure 5. The field emission scanning electron microscope (FESEM) image from amorphous SiO_2 Nanoparticle synthesized.

As seen in Figure 5, silicon Nanoparticles can be obtained by the environmentally friendly short method (EFSM) method of RHs ball-milled precursor. The FESEM microscope has a capability named the chemical analysis energy-dispersive X-ray spectroscopy (EDS). By using EDS determined the composition of the synthesized Nanoparticle. The results of the EDS test are shown in Figure 6.

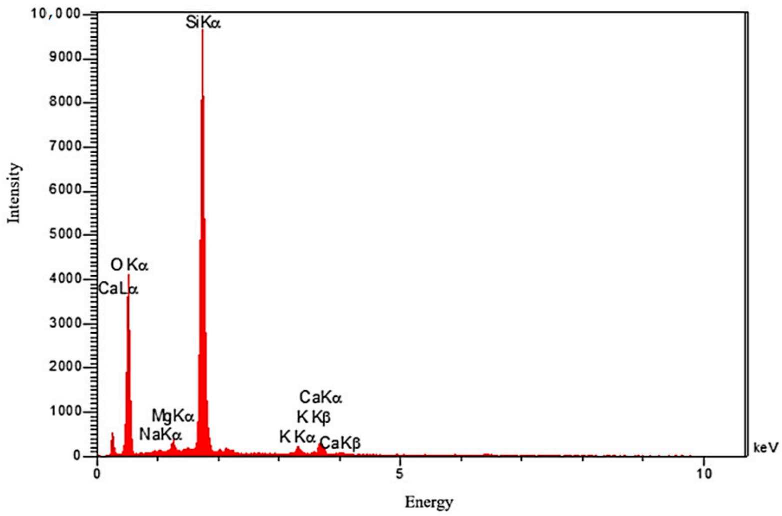

Figure 6. Analysis of EDS.

In Figure 6, it can be observed that the highest peak related to the silicon atoms and oxygen atoms in the second peak appeared. The third peak is the golden atom with which the silica Nanoparticles are coated. The atomic values obtained from the EDS test presented in Table 2 are per weight percentage. Using X-ray fluorescence (XRF), we determined the purity of the synthesized Nanoparticles on the produced powder. The results of the XRF test are presented in Table 3. According to the results of Table 3, nanoparticles were synthesized with a purity of 94.5%, which indicates the method conducted has good efficiency.

Table 2. Results of the EDS analyses.

Components	Si	O	K	Ca	Mg	Na
Weight %	33.96	60.61	1.41	2.48	1.14	0.04

Table 3. Results of XRF analyses.

Composition	SiO_2	P_2O_5	Fe_2O_3	K_2O	CaO	Cl	Na_2O	Al_2O_3	TiO_2	L.O.I	La&Lu
Weight %	94.5	0.39	0. 36	0.66	1.1	0.17	N.D	N.D	0.01	2.05	<1

For precisely determining the size of the NPs, an image was taken of the Nanoparticles by using transmission electron microscopy (TEM). The results of this analysis are shown in Figure 7. As can be seen in Figure 7, the synthesized Nanoparticles have dimensions less than 100 nm.

RH has a high content of silica in its structure compared to other plant waste. The efficiency of Nanoparticles synthesized in this research was 24% by weight. A total of 67 gr of Nanoparticles was derived from 278 gr of precursor material by the introduced synthesis method. By using the planetary ball-milling machines, before the calcination stage, raw materials are reduced in size of from millimeters to micrometers, which results in the available surface area or specific surface area (SSA) being increased.

Milling raw materials causes a promotion of the amount of heat absorption into the carbon, which increases the carbon oxidation before it reaches the dissociation temperature of K_2O. If the carbon cannot be oxidized from the structure of the pre-material before reaching the temperature of the thermal decomposition of the potassium oxide (346.85 °C) and its conversion to potassium, the K (melting point 63.50 °C) in the RHs causes surface melting of the silica and carbon gets entrapped in the melt. When carbon is trapped in a potassium-rich melt, it cannot be oxidized because it is not in direct contact with air.

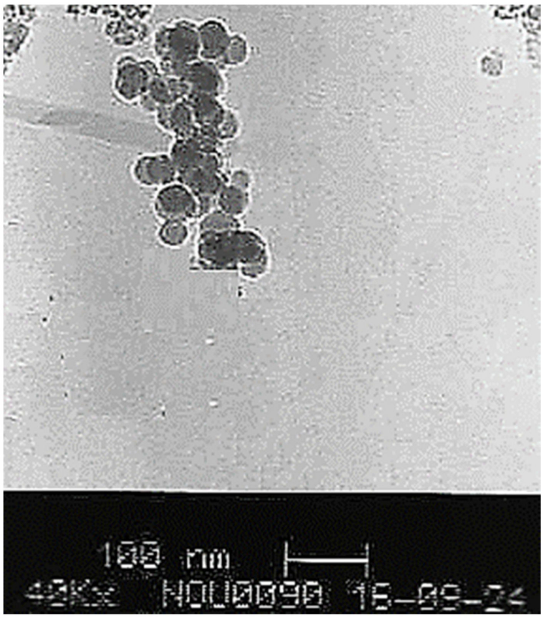

Figure 7. The transmission electron microscopy (TEM) image of the silicon oxide Nanoparticles depicting the uniformity of the particle distribution. These particles appeared to have the dimensions of <100 nm.

Using a ball-mill after the calcination, due to that the cell and ball is in contact with the silica, the cell wall will be eroded and the amount of iron will increase in the product, which requires the use of chemical treatment to remove it. In contrast, by placing it in the pre-calcination stage, the amount of iron entering the pre-material is reduced in the pyrolysis stage. As can be seen in Table 3, the purity of the Nanoparticles without the use of any chemical treatment is 94.5%. According to Figure 5, the structure of the synthesized Nanoparticles is amorphous and the dimensions are below 100 nm.

The calcination temperature and heating rate in the synthesis process have a significant effect on the shape and size of Nanoparticles. A sudden rise in heat rate in the process of thermal decomposition does not allow complete oxidation of organic matter so that it will increase the number of impurities in the final product. Temperatures above 600 °C gradually cause fused silica Nanoparticles that will result in (above 700 °C) increasing in size or deforming from amorphous to crystalline shape. With temperatures less than 600 °C for 5 h, the process is not able to oxidize carbon completely, and some organic materials appear in the fixed carbon in the final product.

In (2015), Ghorbani et al. carried out a study about the synthesized amorphous silica nanoparticle from rice husk ash (RHA) with the use of chemical materials to which the results are similar to the EFSM method. They initially treated RH with a variety of acids such as H_2SO_4, HNO_3, and HCl then calcination occurs at 600 °C for six h to produce RHA with high purity (92.89%, 94.79%, and 95.55%). Then the RHA reacted with sodium hydroxide, producing water-glass. They use sulfuric acid precipitated silica Nanoparticles in a water-glass with a purity of 97%. So, by comparison of the results it can be understood that the EFSM method has a short procedure, low cost for manufacturing, and yield rate with similar results to chemical methods [35].

In another study, Abdullahi et al. (2016) concentrated on the synthesized Nanoparticles by using chemical pretreatment and precursor RH, indicating final results similar to the EFSM technique. The results of the EDS test of Abdullahi et al. on Nanoparticles synthesized by acid leaching are given in Table 4 [36].

Table 4. EDS results of the synthesized silica Nanoparticles by acid treatment [36].

Elements	Average Weight %	
	HCL acid leached	Citric acid leached
Si	45.6	61.9
O	54.4	38.1
Total	100	100

By comparing the results in Tables 2 and 4, it can be concluded that the values of nano-silica obtained (per weight %) in the EFSM procedure and citric acid treatment are similar. Tari et al. (2017) synthesized silica Nanoparticles for their study using chemical material. They used a device similar to the one used in this paper to image from the silica Nanoparticle surface with model MIRA3-TESCAN-XMU. Figure 8, exhibits the photo captured by them. [37].

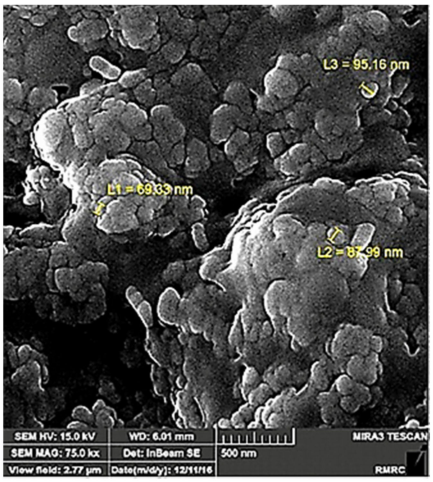

Figure 8. The FESEM image from SiO_2 Nanoparticle synthesized, Reprinted by permission from Springer Nature: International Journal of Environmental Science and Technology, [37] copyright 2019.

By comparing the results in Figures 5 and 8, it can be concluded that the size of nano-silica obtained in the EFSM procedure and chemical method used by Tari et al. (2017) are similar.

4. Conclusions

This paper presents an environmentally friendly short method (EFSM) for the synthesis of amorphous silica oxide Nanoparticles by using rice husks (RHs) agricultural waste. Increasing the special surface area (SSA) of the RHs by planetary ball-milling machines before the calcination stage is able to promote the amount of heat absorption into the carbon in the pre-material, which will increase the carbon oxidation and purification of the silica Nanoparticle. The NPs evaluation was conducted with energy-dispersive spectroscopy (EDS), field emission scanning electron microscope (FESEM), and X-ray fluorescence (XRF). The analysis results indicated the EFSM method was able to synthesize non-toxic amorphous silica Nanoparticles with the purity of 94.5% without using any chemical material. The TEM and FESEM images of the silicon oxide Nanoparticles depict the uniformity of the particle distribution. These particles appeared to have dimensions of <100 nm. The EFSM method has the advantages of being fast and simple to operate, with controllability, great pureness of the resulting Nanoparticle, and low manufacturing cost.

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